Study of The Influence of Cocoon Processing Methods on Fibroin Structure

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Abstract

This paper presents the results of a study examining the effect of cocoon stifling and drying methods on fibroin structure. The paper also presents the technological characteristics of studies on raw silk yields dried using various methods. The study concludes that drying cocoons in a microwave field under specific conditions improves cocoon unwinding.

Keywords: Natural silk, fibroin, sericin, cocoon, stifling cocoons.

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1. Introduction

The growth of production is inevitably accompanied by the development of high-performance processes that ensure the production of finished goods that meet a wide range of demanding consumer demands. Modern textile production relies on such processes and is closely aligned with the scientific and technological advances in the production of natural fibers and threads [1-2].

Despite the rapid development of synthetic materials production, natural silk remains one of the most important natural polymers, with enormous economic significance. Therefore, it is necessary to increase the level of exploration using a wide range of modern physicochemical methods, opening up new avenues for the production and processing of polymers that can improve the performance properties of natural materials and products made from them [3-4].

Various X-ray diffraction and optical methods are used to study the physical properties of substances [5-6]. For example, interplanar spacings in crystals are determined using small-angle X-ray diffraction, the degree of development of the spherulitic structure in polymers is assessed using polarization microscopy, and the parameters of the crystal lattice and its volume are determined using X-ray structural analysis. In recent years, X-ray diffraction analysis methods based on the study of diffraction patterns resulting from the scattering of X-rays by the electron shells of atoms have become increasingly widespread. These methods allow for the precise determination of the crystalline structure and phase composition of the materials being studied. The necessary information is extracted from the analysis of the resulting X-ray diffraction patterns [7-9]. In this study, X-ray diffraction analysis was used to investigate the effect of marinating and drying cocoons on the structure of natural silk. This allows us to assess the impact of this process on the structure of natural silk.

The aim of this study was to investigate structural changes in natural silk exposed to convective drying of cocoons and microwave drying in a TF-HFD-1 cocoon dryer.

X-ray diffraction studies were performed using a DRON-4 X-ray diffractometer (BSV-11 tube, U = 40 kV, I = 15 mA). Radiation from a copper anode with a wavelength of λ = 1.539 Å, filtered by nickel foil, was used. A SRS-1-0 X-ray spin-coil counter was used as a scattered radiation detector. The method of X-ray reflection from the sample surface was used [10-12]. Samples were used in the form of thin films with varying thermal histories or lightly pressed powders. The dependences of diffracted X-ray intensity on the grazing angle were recorded at angles from 5 to 40°. To study the structure of silkworm cocoons, a piece of cocoon was soaked in water at 20°C for 15 minutes and then straightened under a press. Natural silk fibroin (NSF) was obtained by treating silk fiber three times in a 1% boiling sodium bicarbonate solution, followed by rinsing with distilled water and drying at room temperature using convection and in a microwave field in a TF-HFD-1 cocoon dryer.

Fig. 1 shows X-ray diffraction patterns of the studied cocoon samples with different thermal histories.

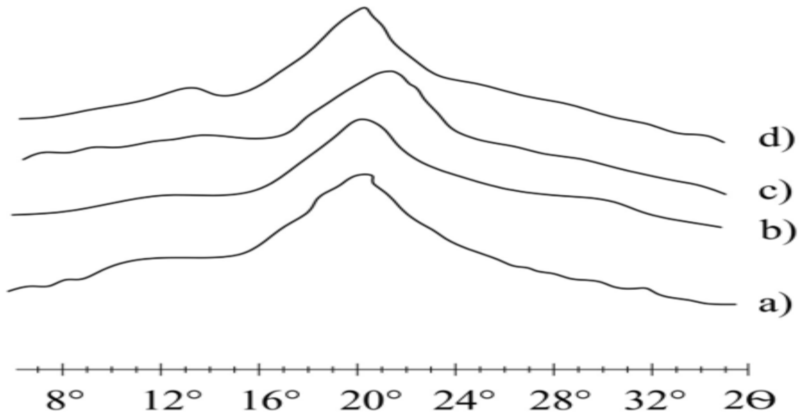

Fig. 1. X-ray photographs of cocoon shells subjected to various treatments;
a - original cocoon; b - cocoon shell dried by convection and c, d - shell of cocoons dried in a microwave field, drying time of cocoons is 10, 20 and 30 min, respectively.

As can be seen from the figure, the X-ray diffraction patterns of the initial samples have one fairly intense reflection at 2θ = 19.50, which is the result of the addition of close reflections 001 and 200 (the interplanar distances d are 4.3 and 4.6 Å, respectively). In addition, there are small rises in the diffraction curve in the region of 2θ = 8-100 and 27-290, which correspond to reflections 100 (d = 9.2 Å) and 210 (d = 3.6 Å). The degree of crystallinity Ck of the initial cocoons, according to the results calculated in our studies, is 51%, which is consistent with the data of other authors [10-12]. Drying live cocoons in a microwave field for 10 min results in a 7% decrease in Ck (Ck = 44%) and a noticeable increase in the half-width of the peak, indicating some disordering of the natural silk structure due to the rapid removal of bound hydration water from the intercrystalline regions. Increasing the microwave drying time to 20 min for the original cocoons does not result in further structural changes. No changes in Ck are observed when drying cocoons of the same batch using convection and in a microwave field, but a slight increase in the intensity of the curve rise in the range 2θ = 11-130 is noted.

Studies have shown that natural silk fibroin has a higher Ck than whole cocoon shells due to the absence of the amorphous component, sericin (Fig. 2). The proportion of crystallinity in fibroin isolated from convective-dried cocoons is 6% lower than that of fibroin treated in a microwave field. As the microwave exposure time of cocoons increases from 10 to 20 minutes, the proportion of crystalline regions in natural silk fibroin increases. Furthermore, X-ray diffraction patterns of both samples show a noticeable shift in the reflex from 20.2 to 19.50, indicating changes in the crystal lattice.

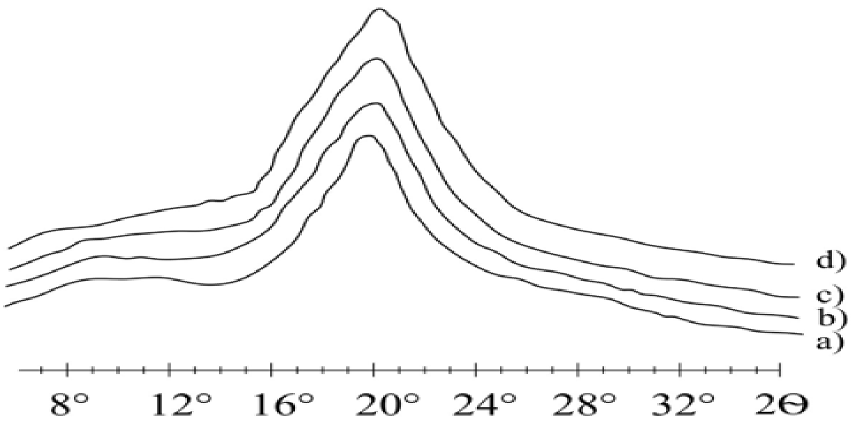

Fig. 2. X-ray diffraction patterns of natural silk fibroin isolated from cocoons dried in a microwave field (a, b, c) and convectively (d).
Drying time, min: a-10 min; b-20 min; c-30 min; d-3 hours.

To study the onset of structural changes in cocoon shells during prolonged drying at high temperatures, silk fiber samples were processed in a high-temperature chamber. X-ray diffraction patterns were recorded as the temperature increased at 20, 100, and 120°C. The samples were then kept at 120°C for 5 hours, recording X-ray diffraction patterns every hour (table).

Increasing the heating temperature of the fiber in a stretched state to 120°C initially leads to a decrease in crystallite size (a shift in the reflection toward larger angles from 2θ = 20.20° at 250°C to 20.80° at 120°C) and a gradual increase in the proportion of crystalline regions. All this may indicate some disruption of the packing of the polypeptide chains with increasing temperature. Maintaining the fiber at 120°C for more than 2 hours (probably due to an increase in interplanar distances) leads to a shift in the peak toward smaller angles, coarsening of the crystallites, and a slight decrease in the Ck of the sample by an amount coinciding with the degree of crystallinity of silk fibroin treated with the convective method.

Table 1.
Characteristics of structural changes in natural silk fibers during heat treatment

Indicator	Temperature, ^{0}C		
	25	100	120
Crystallite size, Å	25	20	22
Degree of crystallinity, %	46	56	58

A study was also conducted to examine the effect of the drying process on the layers of cocoon shells. For this purpose, X-ray photographs were taken of different layers of the cocoon, both the original and those boiled in a soda solution to free fibroin from sericin.

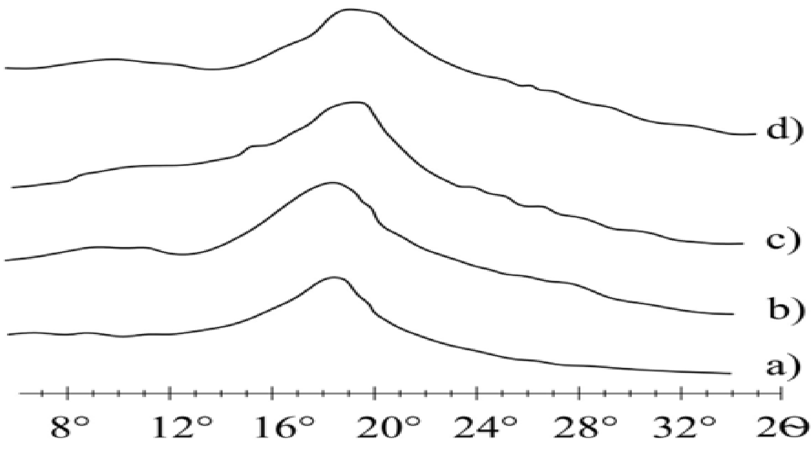

Fig. 3. X-ray photographs of cocoon shells obtained by convective drying: a - original whole cocoon; b - from the outer; c - middle and d - inner layer of the cocoons.

X-ray diffraction patterns of the various layers of the cocoon shell show virtually no difference in crystallinity. The Ck of the outer layer of the original cocoon is 58%, while all other layers have a Ck of 57%. Crystallite sizes remain constant and correspond to 20 Å (Fig. 3). In natural silk fibroin, the proportion of crystalline regions varies somewhat across the layers. The degree of crystallinity of the outer layer (65%), although slightly different from that of the inner layers (68%), is also slightly smaller (20 Å versus 21 Å). This can apparently be attributed to various factors, such as the influence of light, air, and temperature during convective drying on the outer layer. The lower Ck values in the inner layers can obviously be explained by the natural properties of the cocoon shell and the proximity of the inner layers to the pupa itself.

In studies [14-15], for example, it is described that the proportion of crystalline regions in the inner layer of the cocoon decreases due to the larger number of amorphous regions. When comparing X-ray diffraction patterns of natural silk fibroin obtained by convective methods from different layers of the cocoon, a shift in the peaks of the maxima is noted, associated with different structural ordering in different layers of the cocoon (Fig. 4).

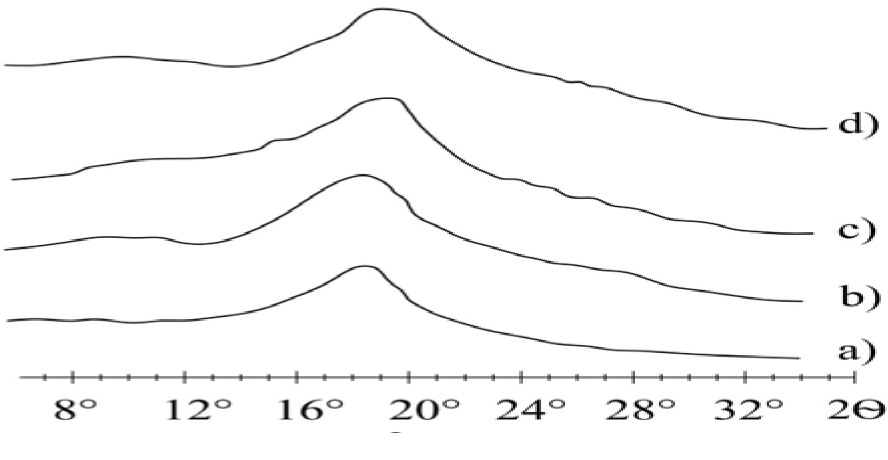

Fig. 4. X-ray images of natural silk fibroin obtained: a - from a whole cocoon; b - from the outer; c - from the middle second layer; and d - from the inner layer of the cocoon shells.

The samples studied, specifically those from cocoons dried using various methods, were unwound, and the raw silk yield was calculated. The studies showed that drying the cocoons in a microwave field for 20 minutes resulted in improved unwinding compared to unwinding them using convective methods; the raw silk yield in the former case increased by 0.93%. In this case, the increase in the yield of raw silk during the unwinding of cocoons dried in a microwave field can be characterized by the strengthening of natural silk fibers due to an increase in the proportion of crystalline regions, therefore, the strength of the fibers is directly dependent on this characteristic [14-15].

Based on the presented X-ray structural analysis studies, it can be concluded that during the process of drying cocoons in a microwave field, not only the time of their marinating and drying is reduced, but also some positive changes occur in the structure of natural silk fibroin.

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